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Development of antibacterial knitted fabrics from polyester-silver nanocomposite fibres

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Development of durable antibacterial knitted polyester fabrics by incorporation of small proportion of polyester-silver nanocomposite fibres has been attempted in this research. Polyester-silver nanocomposite fibres are blended with normal polyester fibres at three weight fractions (10, 20 and 30%). Three parameters, namely blend proportion (wt.%) of nanocomposite fibres, yarn count and knitting machine gauge, have been varied for producing 27 knitted fabrics. Knitted fabrics prepared from polyester-silver nanocomposite fibres show good to excellent antibacterial activity (65-99%) against both *S. aureus* and *E. coli* bacteria. Blend proportion of nanocomposite fibres is found to be the most dominant factor in influencing the antibacterial activity of knitted fabrics. Antibacterial activity also increases with coarser yarn count and higher knitting machine gauge. It is also found that the tightness or compactness of knitted fabrics is correlated positively with their antibacterial activity.

Keywords: Antibacterial activity, Knitted fabric, Nanocomposite fibres, Polyester, Silver nanoparticles

1 Introduction

Textiles materials have long been recognised as media which support the growth of microorganisms, such as bacteria and fungi. These microorganisms are found almost everywhere in the environment and can multiply quickly when basic requirements, such as moisture, nutrients, and temperature are met. Polyester is a versatile manmade fibre and finds use in a variety of applications, such as apparel, industrial fabrics, etc. It is the highest tonnage fibre being produced and used all over the world. Manufacturing of sportswear, medical and protective textiles based on polyester fabrics has continually increased over the last few decades. Natural fibres, which are mostly cellulosic or protein-based in nature, can act as food for many microorganisms, especially in warm and humid conditions. They can be attacked by microorganisms, resulting in degradation, discolouration and malodour generation. Growth of microbes is slower on synthetic fibres. However, these fibres can still act as a shelter for microbes where they can survive and grow. Foot infection, for example, has been found to be more pronounced in synthetic fibre socks than natural fibre socks. Hsieh and Merry¹ observed that adherence of bacteria on to

fabric increases with increase in polyester fibre content. Hence, in certain situations where a large number of people from very diverse backgrounds and medical conditions come in contact with each other, such as hospitals, schools, and colleges, cultural and religious functions, etc., textiles can become a potent source of cross infections¹⁻⁸.

Silver is one of the most widely used antibacterial agents since the evolution of the mankind. It is known that silver is one of the safest antibacterial agents as compared to organic compounds⁹. Silver has been medically proven to kill over 650 disease-causing organisms¹⁰⁻¹². Silver ions rapidly kill microbes by blocking the cell respiration pathway or breaking the outer cell wall. The speed of action is almost instantaneous once the silver ion reaches the microbe¹³. The efficacy of microbe killing is dependent on the amount of silver ions present, which, in turn, is governed by the size of silver particles present in the product. Because of their very high specific surface area, silver nanoparticles have garnered extensive attention both commercially and in research. Nano-silver has found a wide range of applications in dressings for burns, scald and acne^{12, 14-16}.

Jeong *et al.*¹¹ reported the incorporation of the nano- (100 nm) and micro- (1 μ m) silver powders in the nanocomposite fibres prepared by direct melt-compounding. The quantitative antibacterial

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tests showed that the fibres incorporated with nano-silver powder exhibited better antibacterial activity than the fibres incorporated with micro-silver powder. Chen *et al.*¹⁷ discussed the preparation of silver-acrylonitrile-vinylidene chloride (Ag-AN-VC) copolymer fibres by spinning route. These fibres were tested against both Gram-positive *Staphylococcus aureus* (*S. aureus*) and Gram-negative *Escherichia coli* (*E. coli*) bacteria and it was found that fibres containing 20 wt.% Ag-AN-VC showed over 99% bactericidal efficiency for both the bacteria. Erem *et al.*¹⁸ studied the antibacterial activity of silver nanoparticle loaded polyamide 6 (PA6) nanocomposite fibres with different percentage of silver against *Klebsiella pneumonia* and *S. aureus* bacteria. The results showed that the nanocomposite fibres were effective against both the bacteria and their efficacy increased with increasing nanoparticle content. Yeo and Jeong¹⁹ reported that antibacterial activity of polypropylene/silver nanocomposite fibres can be enhanced if the nano-silver part is confined to the sheath section of fibres. Perelshtein *et al.*²⁰ reported the preparation of nylon, polyester and cotton with antibacterial properties by deposition of silver nanoparticles on fabrics using sonochemical method. The physical and chemical analyses showed that nanocrystalline pure silver (80 nm size) is finely dispersed on the fabric's surface without any significant damage to structure of yarn. The coating was stable on the fabric for at least 20 washing cycles in warm (40 °C) water. To obtain better adhesion between fabrics and silver nanoparticles, polyester fabrics were given a pre-treatment by Corona plasma¹⁵. As a result of this pre-treatment, antibacterial activity of the treated polyester fabrics improved.

Lin *et al.*²¹ incorporated silver particles into hollow polyester fibres. These particles were evenly adsorbed into the inner surface of fibres and silver ions were released into an aqueous medium from the two ends of the fibre. This method precluded the direct contact of silver with body tissues.

The literature review shows that limited research efforts have been made to incorporate silver nanoparticles in fibres at melt spinning stage, whereas most of the efforts revolve around the application of silver nanoparticles on textile substrates through finishing route. Besides, most of the studies on antibacterial nanocomposite fibres are limited to fibre development and their characterisation only. There is a void on development of antibacterial fabrics (woven

or knitted) using such nanocomposite fibres and understanding the role of yarn and fabric parameters on antibacterial activity.

The use of inherent antibacterial polyester fibres by embedding silver nanoparticles into fibres obviates the need for antibacterial finishing. Moreover, it imparts a much more durable antibacterial nature to the fibres. As nano-silver is embedded in the polyester fibre during melt spinning stage, there is no need for any binder to bind the antibacterial agent to the fibre and scarce natural resources like water and energy are also preserved. Moreover, a separate step for application of silver on fibres/fabrics is avoided. Thus, incorporation of silver nanoparticles in fibres through melt spinning is more eco-friendly and sustainable route.

Although silver in any form is a costly additive, it is so potent that very small concentration (400 ppm) is sufficient to impart effective antibacterial (above 90%) functionality to the fibres. The addition of silver at this level causes only 10-12% increase in raw material (fibre) cost. Since the fibre cost is less than 10% cost of a finished product, the overall contribution of silver to the final product cost is around 1%, which can be easily compensated by value addition for the durable antibacterial activity.

Thus, in this research work, an attempt has been made to impart antibacterial functionality to 100% knitted polyester fabrics by blending polyester-silver nanocomposite fibres with normal polyester fibres at various blend proportions and to study the effect of yarn and fabric construction parameters on antibacterial efficacy of such fabrics.

2 Materials and Methods

2.1 Materials

Nano-silver impregnated polyester staple fibres (38 mm, 1.35 denier and nano-silver loading of 400 ppm) and normal polyester fibres (38 mm and 1.4 denier) were used in this research for yarn preparation. Ethanol (99.9% pure, Changshu Yangyuan Chemical, China) was used as-received without any further purification. Luria-Bertani, LB media and agar-agar were procured from Himedia laboratories Pvt. Ltd., India.

2.2 Sample Preparation

Polyester yarns were spun by keeping the proportion of polyester-silver nanocomposite fibres at three levels (10, 20 and 30%). Normal polyester fibres constituted the remaining 90, 80 and 70%

respectively. Three yarns of varying counts (20^s Ne or 29.5 tex, 30^s Ne or 19.7 tex and 40^s Ne or 14.8 tex) were produced. Thus, a total of nine yarns of 100% polyester were produced. The yarns were plied (2 ply) for ease of operation on single jersey weft knitting machine. Three parameters, namely blend proportion (wt.%) of nanocomposite fibres, yarn count and knitting machine gauge were varied to obtain 27 knitted samples as shown in Table 1. The samples were relaxed by immersing in a water bath, containing 0.01% wetting agent at 38°C for 12 h followed by gentle hydro-extraction and drying on a flat surface for 24 h at ambient conditions.

2.3 Characterisation of Nanocomposite Fibres

The surface characteristics of nanocomposite and normal polyester fibres were studied with the help of scanning electron micrograph or SEM (ZEISS, model: EVO 50). The samples were first coated with a gold layer to provide surface conductivity before scanning. Energy dispersive X-ray (EDX) was carried out to verify the presence of silver in polyester fibre.

The X-ray diffraction (XRD) pattern were recorded

Table 1 — Antibacterial activity of knitted materials

Sample No.	Blend proportion, %	Yarn count Ne	Machine gauge inch ⁻¹	Bacterial colony reduction, %	
				<i>S. aureus</i>	<i>E. coli</i>
1	10	2/20	12	71.88	73.13
2	20			82.50	85.82
3	30			88.75	91.79
4	10	2/30		68.42	67.56
5	20			81.11	84.86
6	30			85.00	88.10
7	10	2/40		66.67	63.20
8	20			71.05	73.11
9	30			80.00	82.54
10	10	2/20	16	83.64	87.61
11	20			94.55	96.19
12	30			96.36	98.09
13	10	2/30		71.67	73.84
14	20			85.83	86.15
15	30			93.33	92.30
16	10	2/40		63.70	65.00
17	20			76.30	77.14
18	30			84.44	83.80
19	10	2/20	20	86.67	85.71
20	20			95.56	97.14
21	30			98.89	98.57
22	10	2/30		79.09	75.71
23	20			86.36	87.14
24	30			96.36	94.28
25	10	2/40		73.08	79.5
26	20			84.62	85.00
27	30			92.31	93.50

using a Philips Analytical X-ray Instrument, X'Pert-Pro (PW 3040 and PW 3060 control unit) with CuK α radiation (wavelength 1.54056 Å). Diffractogram of normal polyester and polyester-silver nanocomposite fibres were recorded at a scanning speed of 10°/min and a chart speed of 5 mm/min. Transmission electron microscopy (TEM) was performed to determine the morphology, size and shape of silver nanoparticles. TEM measurements were done on JEM-1400 Plus, operating at 120 kV. The TEM grid was prepared by placing a drop of silver nanoparticle dispersion on it. The silver nanoparticle dispersion was prepared by dissolving the polyester part of polyester-silver nanocomposite fibres in 50% (w/v) sodium hydroxide solution at 27 °C in 30-60 min. After neutralisation with acetic acid and washing with ethanol in a Thermo fisher sorvall ST 8 centrifuge machine with 7500 rpm, the dispersion was placed on a carbon-coated copper grid and allowed to dry under ambient conditions.

2.4 Determination of Antibacterial Activity

Knitted fabrics were subjected to quantitative AATCC 100-2004 colony counting test to evaluate their antibacterial activity. Gram-positive bacteria (*S. aureus*) and Gram-negative bacteria (*E. coli*) were used as the test bacteria. A homogeneous mixture of bacteria (known as mother culture) was prepared by mixing luria broth (nutrient) with deionised water and incubating it at 37°C for 24 h. Knitted fabric samples (3 cm × 1 cm) were placed in test tubes. Test tube without fabric sample was considered as control. The test tubes were inoculated with luria broth culture having a bacterial concentration of 2 × 10⁵ colony forming units/mL (CFU/mL) and incubated at 37°C for 24 h. After incubation, 1 mL of bacterial solution was taken in a test tube and serial dilutions were made with deionised water. A 100 µL suspension of the last dilution was poured on agar plates and incubated at 37°C for 24 h. After incubation, the numbers of colony forming units were counted using the colony counter and the antibacterial activity was calculated using the following equation:

$$R(\%) = \frac{B - A}{B} \times 100 \quad \dots (1)$$

where *R* is the percentage reduction; *A*, the number of bacteria recovered from the inoculated test specimen swatches incubated over the desired contact period; and *B*, the number of bacteria recovered from the

control specimen swatches incubated over the desired contact period.

3 Results and Discussion

3.1 Characterisation of Nanocomposite Fibre

The surface morphology of the normal polyester and polyester-silver nanocomposite fibres was analysed using SEM as shown in Fig. 1. Higher surface roughness, which can be attributed to the presence of nano-silver, has been observed in case of polyester-silver nanocomposite fibres as compared to normal polyester fibres. The presence of silver in the nanocomposite fibre is also confirmed by EDX spectra. The EDX results show that around 0.84% silver is present at the surface of polyester-silver nanocomposite fibres (Table 2). The shape and size of the resultant particles are elucidated with the help of TEM (Fig. 2). The TEM micrographs suggest that the size of the particles is around 20 nm. The particles are

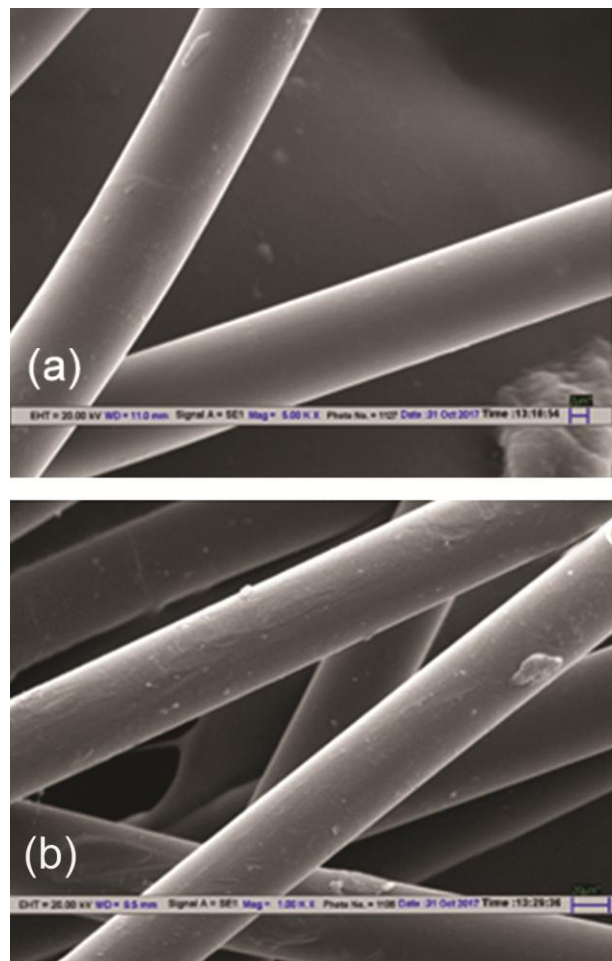


Fig. 1 — SEM images of (a) normal polyester fibre ($\times 5000$, Scale bar 2 μm) and (b) polyester-silver nanocomposite fibre ($\times 1000$, Scale bar 20 μm)

found to be roughly spherical in shape. The XRD patterns of normal polyester and polyester-silver nanocomposite fibres are shown in Fig. 3. It is observed that there is no significant difference in the XRD patterns of normal polyester and polyester-silver nanocomposite fibres. This implies that the incorporation of nano-silver into the polyester fibres,

Table 2 — EDX results of polyester-silver nanocomposite fibre

Element	Atomic number	Weight, %	Atomic, %
C	6	52.96	60.36
O	8	46.20	39.53
Ag	47	0.84	0.11

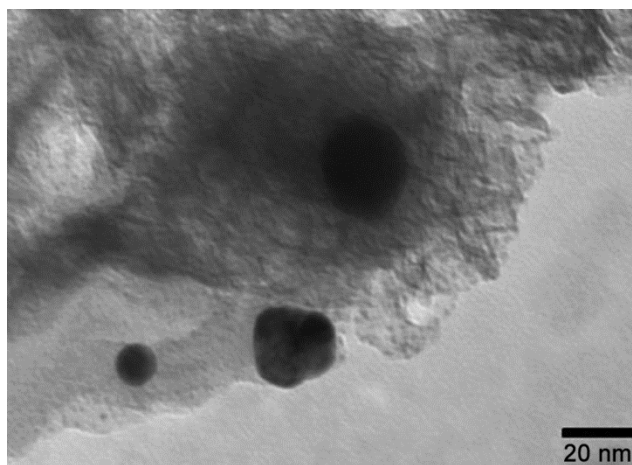


Fig. 2 — TEM image of silver nanoparticles

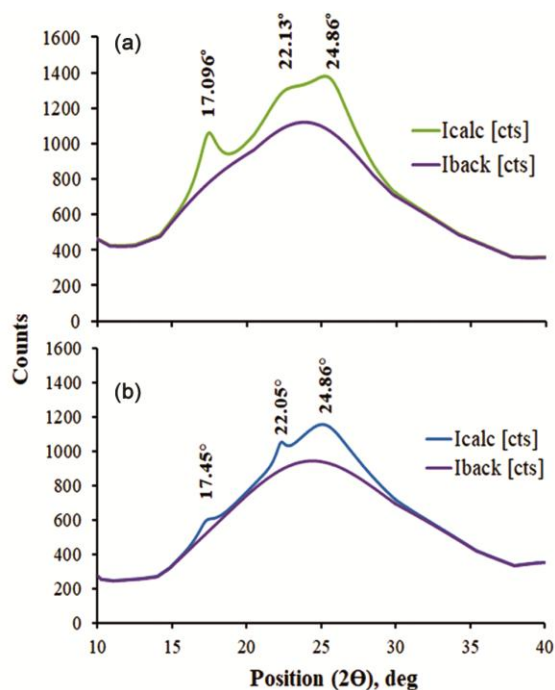


Fig. 3 — X-ray diffraction pattern of normal polyester fibre (a), and polyester-silver nanocomposite fibre (b)

during melt spinning, has not affected the crystalline structure of the fibre.

3.2 Antibacterial Activity of Knitted Fabrics

Antibacterial activity of knitted fabrics against *S. aureus* and *E. coli* bacteria was examined and the outcomes are shown in Table 1. It is observed that knitted fabrics made from nanocomposite fibres have very good activity against both *S. aureus* and *E. coli* bacteria (63.20-98.89%). It is also observed that the blend proportion of nanocomposite fibres, yarn count and machine gauge influence the antibacterial activity of knitted fabrics. The parameters studied in this work can be seen to influence the antibacterial activity of fabrics by up to 35%. The bacterial colony reduction increases rapidly with increase in the blend proportion of nanocomposite fibres as shown in Table 1.

3.3 Effect of Blend Proportion and Yarn Count on Antibacterial Activity

The effect of blend proportion (wt.%) and yarn count, keeping the machine gauge constant at 16 inch⁻¹, on antibacterial activity against *S. aureus* and *E. coli* is shown in Figs 4(a) and (b) respectively. As the blend proportion of nanocomposite fibres increases,

the antibacterial activity increases consistently. On the other hand, antibacterial activity reduces as the yarn becomes finer. Keeping the yarn count and machine gauge constant at 2/30s Ne and 16 inch⁻¹ respectively, the increase in blend proportion of nanocomposite fibres from 10 to 20 and finally to 30% leads to an increase in antibacterial activity from 71.67 to 85.83 and finally to 93.33% against *S. aureus* bacteria [Fig. 4(a)]. The corresponding values for *E. coli* are 73.84, 85.15 and 92.3% [Fig. 4(b)]. The impact of blend proportion of nanocomposite fibres on antibacterial activity is found to be similar for other yarn counts (2/20^s and 2/40^s) as depicted in Figs 4(a) and (b). As the blend proportion increases, more Ag⁺ ions interact with the O⁻ antigens of the bacteria, which, in turn, disrupt the cell wall of bacteria thus killing it.

It is also observed from Figs 4(a) and (b) that knitted fabrics with coarser yarns demonstrate higher antibacterial activity. At the same blend proportion, the number of polyester silver nanocomposite fibres in a coarser yarn would be higher, which means more surface area of inherently antibacterial fibres is available for higher antibacterial activity.

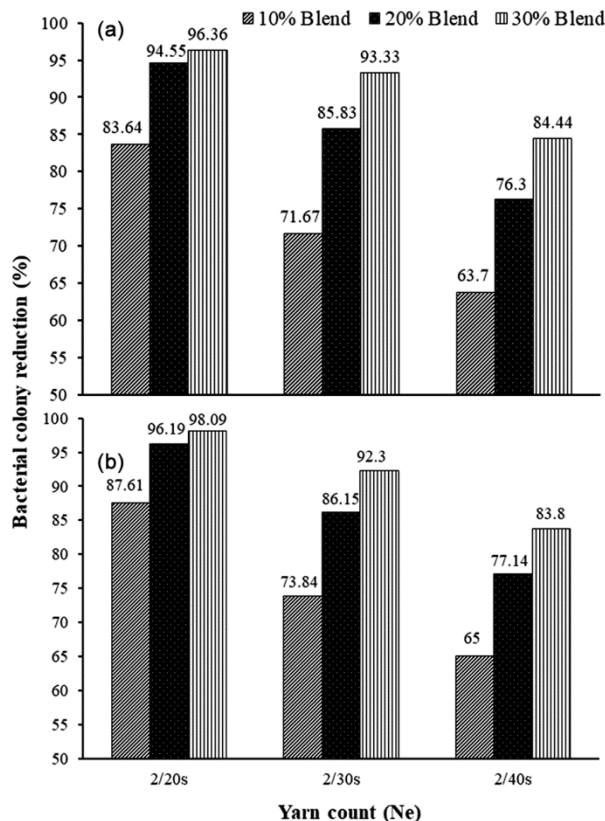


Fig. 4 — Effect of blend proportion on bacterial colony reduction against *S. aureus* (a), and *E. coli* (b)

3.4 Effect of Knitting Machine Gauge on Antibacterial Activity

Effect of machine gauge and yarn count, which decide the tightness or compactness of knitted fabric, on the antibacterial activity of knitted fabrics having 20% blend proportion of nanocomposite fibres, against *S. aureus* and *E. coli* bacteria are shown in Figs 5(a) and (b) respectively. When machine gauge increases from 12 to 16 and then to 20 inch⁻¹, keeping the blend proportion of nanocomposite fibres and yarn count constant at 20% and 2/30^s Ne respectively, the antibacterial activity increases from 84.86 to 86.15 and finally to 87.14% for *S. aureus* and from 81.11 to 85.83 and 86.36 % for *E. coli*.

Generally, the tightness factor of single jersey knitted fabric is expressed using the following equation:

$$\text{Tightness factor} \propto \frac{\text{Yarn diameter}}{l} \quad \dots (2)$$

The loop length (l) was measured by unraveling the courses from knitted fabrics and the yarn diameter was measured using optical microscope. Then relative tightness factor was calculated using Eq. (2).

Tightness factor is the ratio of area covered by yarn to the area of repeating unit. It represents the

compactness or looseness of the material. Tightness factor of material increases with increase in machine gauge, which results in formation of large number of smaller loops. As the tightness factor of the knitted fabric increases, more nanocomposite fibre surface becomes available per unit area for bacterial inhibition. Thus, the contact between nanocomposite fibres and bacteria increases, leading to increased antibacterial activity.

Figure 6(a) and (b) depict the influence of tightness factor on antibacterial activity at different blend proportions of nanocomposite fibres, against *S. aureus* and *E. coli* respectively. Tightness factor of knitted fabrics shows good association with antibacterial activity. It demonstrates that, for a given blend proportion of nanocomposite fibres, antibacterial activity increases with the increase in tightness factor or compactness of knitted fabrics.

3.5 Statistical Significance of Parameters

Table 3 presents the analysis of variance (ANOVA) results. The main effects of blend proportion of nanocomposite fibre, yarn count and machine gauge are found to be statistically significant at 99% level ($p < 0.0001$). Table 4 shows the % contribution of three parameters, based on the sum of

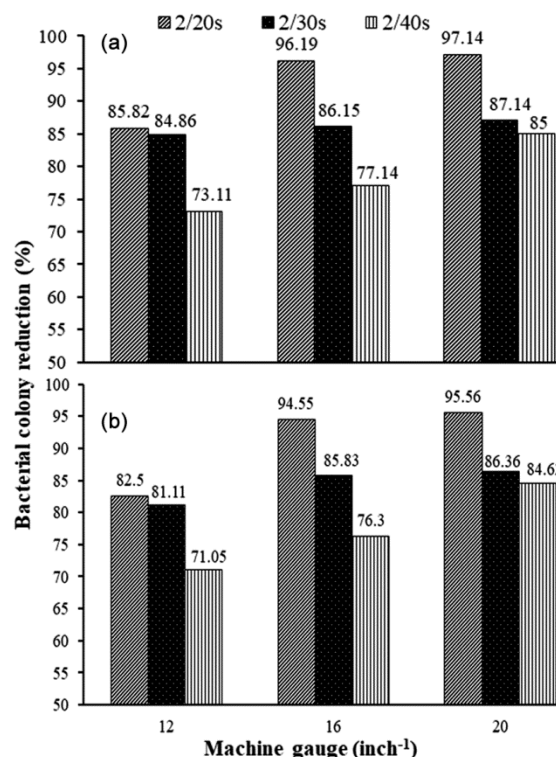


Fig. 5 — Effect of yarn count and machine gauge on bacterial colony reduction against *S. aureus* (a) and *E. coli* (b)

squares values reported in Table 3, to antibacterial activity. Blend proportion contributes 49.63% and 50.02%, whereas yarn count contributes 23.63% and 26.13%, towards antibacterial property against *S. aureus* and *E. coli* respectively. Clearly, blend proportion plays a dominant role in influencing antibacterial activity as compared to yarn count and machine gauge.

The linear regression models for predicting average antibacterial activity against *S. aureus* and *E. coli* are represented by the following equations:

S. aureus

$$\text{Antibacterial activity (\%)} = 61.96 + 0.84A - 1.17B + 1.36C \quad [R^2 = 0.91] \quad \dots (3)$$

E. coli

$$\text{Antibacterial activity (\%)} = 66.43 + 0.84A - 1.24B + 1.20C \quad [R^2 = 0.88] \quad \dots (4)$$

where *A*, *B* and *C* are the blend proportion of nanocomposite fibres (%), yarn count (Ne) and machine gauge (inch⁻¹) respectively. The model is statistically significant with a very small *p*-value. The adjusted coefficient of determination is found to be

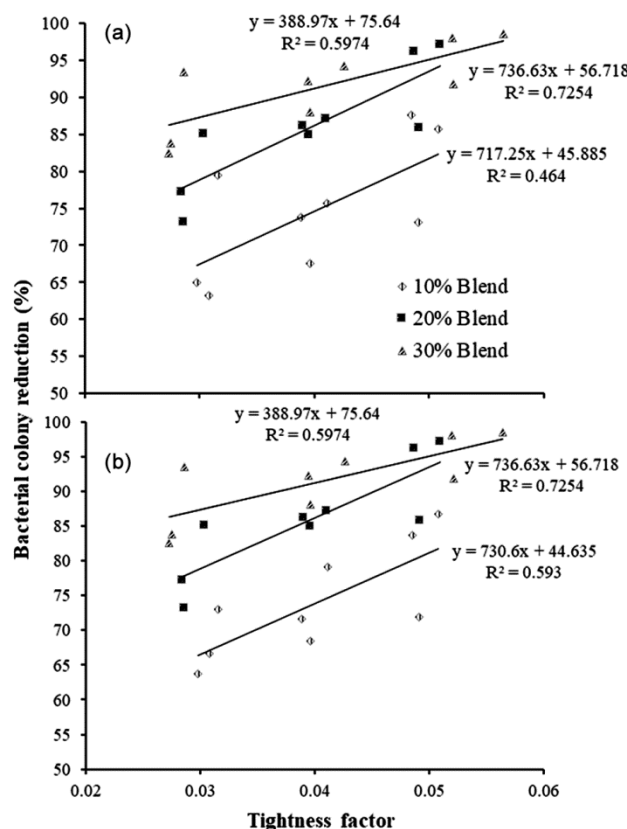


Fig. 6 — Influence of compactness of knitted fabrics on antibacterial activity against *S. aureus* (a), and *E. coli* (b)

Table 3 — Analysis of variance (ANOVA) for antibacterial activity

Source of variation	Sum of squares	Degrees of freedom	Mean square	F-value	p-value
<i>S. aureus</i>					
Model	2426.01	6	404.33	50.35	< 0.0001
Blend proportion (A)	1283.69	2	641.85	79.93	< 0.0001
Yarn count (B)	611.17	2	305.58	38.05	< 0.0001
Machine gauge (C)	531.15	2	265.57	33.07	< 0.0001
Residual	160.61	20	8.03		
Cor total	2586.61	26			
<i>E. coli</i>					
Model	2438.14	6	406.36	38.02	< 0.0001
Blend proportion (A)	1326.58	2	663.29	62.05	< 0.0001
Yarn count (B)	693.04	2	346.52	32.42	< 0.0001
Machine gauge (C)	418.52	2	209.26	19.57	< 0.0001
Residual	213.77	20	10.69		
Cor total	2651.91	26			

Table 4 — Contribution (%) of parameters on antibacterial activity

Parameters	<i>S. aureus</i>	<i>E. coli</i>
Blend proportion (A)	49.63	50.02
Yarn count (B)	23.63	26.13
Machine gauge (C)	20.53	15.78

around 0.91 and 0.88 for *S. aureus* and *E. coli* respectively.

4 Conclusion

Knitted fabrics prepared by polyester yarns containing small proportion (10-30%) of polyester-silver nanocomposite fibres demonstrate very good (63.20-98.89%) antibacterial activity against both Gram-positive (*S. aureus*) and Gram-negative (*E. coli*) bacteria. Antibacterial activity increases rapidly with increase in blend proportion of nanocomposite fibres. At 30% blend proportion of nanocomposite fibres, knitted fabrics demonstrate at least 80% bacterial colony reduction against both the bacteria which improves further with coarser yarn count and

higher knitting machine gauge. The tightness factor of knitted fabrics shows good association with the antibacterial activity. Statistical analysis reveals that all three parameters (blend proportion of nanocomposite fibres, yarn count and machine gauge) are significant at 99% confidence level. Blend proportion of nanocomposite fibres is found to be the most dominant factor in influencing the antibacterial activity of knitted fabric followed by yarn count and machine gauge.

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